FINAL REPORT

Project Title: Enhancing YBCO performance through fundamental process evaluation and characterization

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Project Objective: *The main goal of the program is to develop a fundamental understanding of the mechanisms that result in defects when thick YBCO films are prepared on multi-layered buffered, metal substrates*.

The scope of this program has evolved from studying the fundamental processes that lead to defect formation to incorporation of suitable defects for enhancing flux pinning properties. Following are the issues that were addressed:

a) Comparison of YBCO deposition processes including use of aerosol-assisted deposition (AAD) in developing defect free superconductor films.
b) Establish the relationship between film microstructure, defects, thickness, YBCO deposition process and critical current density (J_c).
c) Investigation of feasibility of using laser striation method for reducing the ac losses in YBCO coated conductors. Study of relationship between film microstructure, defects, thickness, YBCO deposition process used and the critical current density (J_c)
d) Investigation of the influence of metal substrate and multi-layer buffer layers on the subsequent growth and performance of thick YBCO layers
e) Investigation of growth mechanism of YBCO deposited using MOD-TFA process
f) Microstructural evaluation of novel buffer layers to develop high current density YBCO coated conductors.

Work Done:

Aerosol assisted deposition technique was developed in house, as an inexpensive non-vacuum technique for deposition of high density, thick YBCO films. The film deposition parameters, precursor composition, heat treatment temperature, pO_2 etc. were optimized for single phase, c-axis oriented film. This process was eventually abandoned due to major scale up issues.

Microstructural and morphological investigation was performed on two multi-layered samples comprising of PLD-YBCO/SRO/H-MgO/ISD-MgO/YSZ/HC (from Argonne National Laboratory- ANL). The first sample carried I_c = 8.4 A, J_c=248 KA/cm^2 while its sister sample (deposited during the same run) showed better performance, i.e ~ 0.9 MA/cm^2. The rest of the layers in the film stack were grown under the same process conditions.

The goal of this study was to delineate the microstructural difference between the high current carrying region and low current carrying region as observed from the magneto optical imaging. The microstructural characterization was performed using Focused Ion Beam (FIB) and Scanning Electron Microscope with Energy Dispersive Spectroscopy attachment (SEM-EDS).

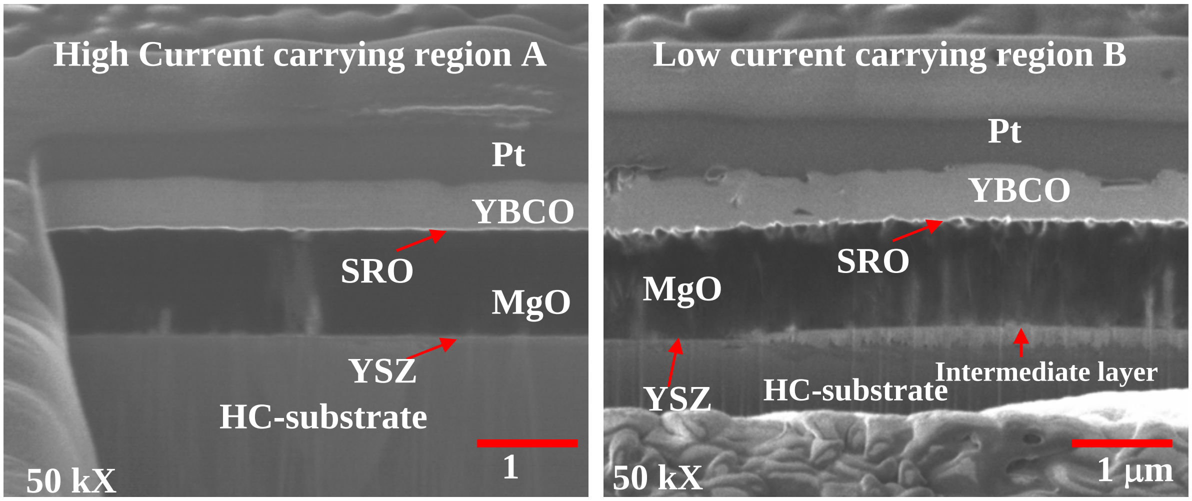

Figure 1: FIB cross sections (tilted at 45^o angle) of high current carrying region and low current carrying region

The YBCO surface morphology of high current carrying region is characterized by essentially uniform matrix with some surface defects (particles ~2-3 mm). Bright field TEM image shows fully dense grains within the YBCO layer. The surface of low current carrying region appears rougher, with black spots (secondary phases). All the layers in high current carrying region are essentially defect free and smooth, with continuous YBCO, SRO, MgO and YSZ layers. Sample curvature is observed in the low current carrying region, with a large number of defects at interfaces. It appears that substrate roughness, and curvature leads to poor quality of YSZ layer, and subsequent transfer of defects to YBCO in low current carrying region.

The feasibility of using laser striation technique for fabrication of YBCO filaments, for reduction of ac losses was investigated in collaboration with Air Force Research Laboratory (AFRL). Three PLD-YBCO samples (YBCO/ YSZ/ HC alloy), were patterned into linear striations by removing strips of the superconductor by laser ablation. Sample 1 was cut in Ar atmosphere while samples 2 (one striation) and 3 (multiple striations) were processed in air. The salient features of the striated architecture is as follows: Width of striation ~ 70 μm. Depth of striated groove ~ 10-20 μm, and the distance between striations in multiple striated sample was ~ 300 μm.

The goal of this work was to investigate the feasibility of laser striation as a route for fabrication of YBCO filaments for reduction of ac losses. Microstructural characterization of PLD-YBCO (YBCO/ YSZ/ HC alloy) filaments and striated region was performed using FIB, SEM-EDS and Rutherford backscattering spectroscopy (RBS) in order to delineate the structural difference due to different processing environments. The microstructural difference between striations in air and argon was delineated.

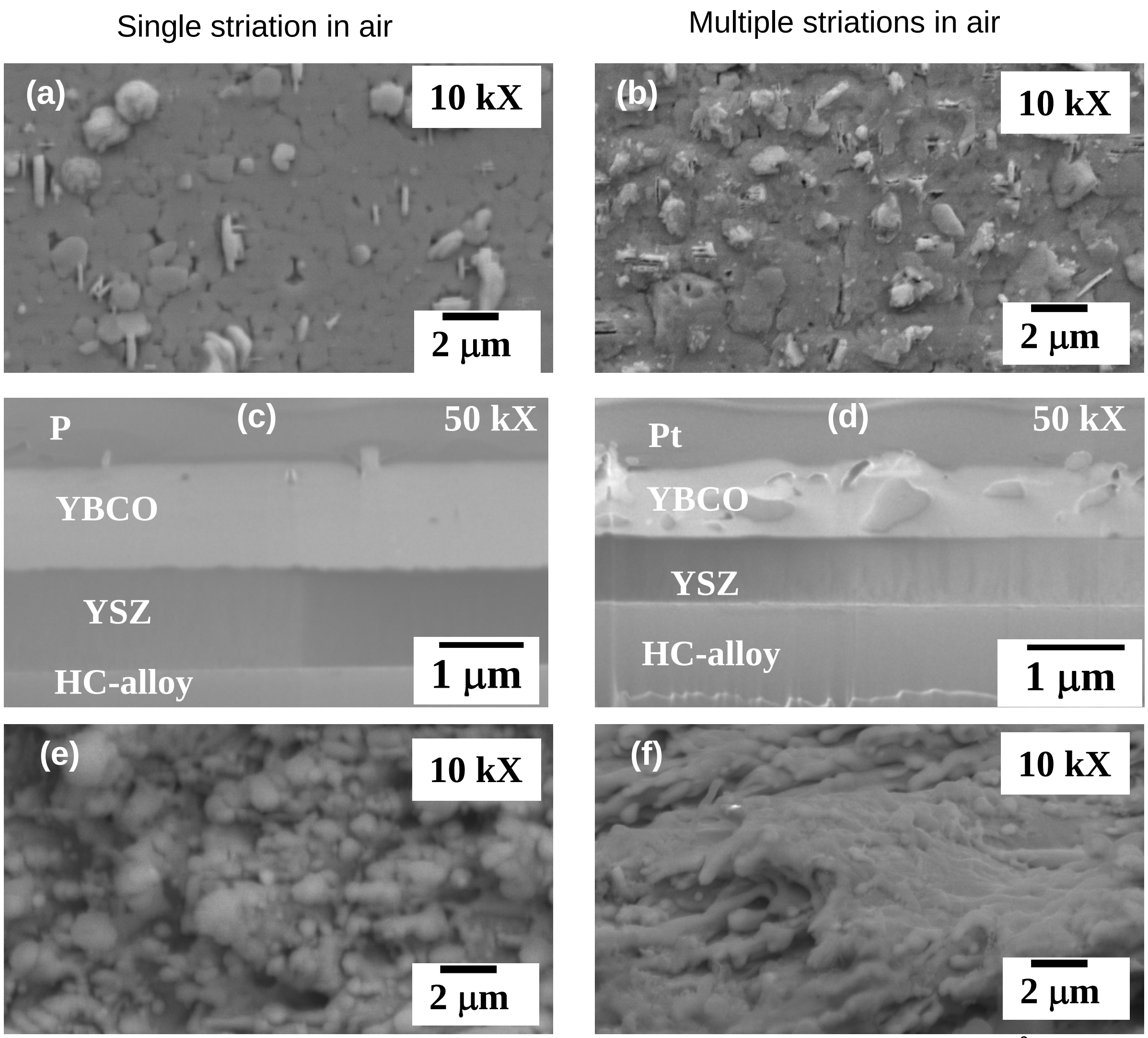

Figure 2: SEM top view outside of striation sample (a) and sample (b); FIB cross section (tilted at 45^o) outside of striation (c) and (d); and SEM top view inside of striation (e) and (f)

In sample with multiple striations, large amount of debris is observed in the non-striated region. Laser ablation appears to melt the material, and the melted surface solidified into globules. At the center of the striation, the surface morphology is very rough and YBCO layer has completely disappeared, moreover, there is no demarcation between YSZ and hastalloy substrate. As per EDS analysis, peaks corresponding to YBCO are observed only in the region outside of striation. Within striated region, the peaks correspond to hastalloy and YSZ.

Multiple striations led to a higher defect density on the YBCO film as compared to YBCO film with a single striation. The debris from laser ablation gets re-deposited on the YBCO surface. The intense heating effect was confirmed by observation of fused globules within the striated layer forming a rope-like structure in the multiply striated sample. In singly striated sample the globules remained discrete.

Feasibility of laser striation technique for creation of YBCO filaments will depend on optimization of filament width and laser intensity.

Albany NanoTech has worked on optimization of the MOD-TFA process in collaboration with American Superconductor Corporation (AMSC). YBCO was deposition using MOD-TFA (TriFluoroAcetate process). The goal is to understand the reaction sequence to achieve high J_c. The coated conductor architecture- YBCO / CeO2 (~50 nm)/ YSZ (~ 350 nm) / Y2O3 (100 nm)/ Ni-NiW. The aim of this investigation is to delineate the mechanism of YBCO formation in the MOD precursors with heat treatment and calcination schedule. In this context, many samples in various stage of processing were investigated for their composition, fluorine content, elemental depth profiling, and cross sectional analysis using techniques such as Rutherford Backscattering analysis (RBS), Nuclear Reaction Analysis (NRA), X-Ray Photoelectron Spectroscopy (XPS), and FIB and Transmission Electron Microscopy (TEM) respectively.

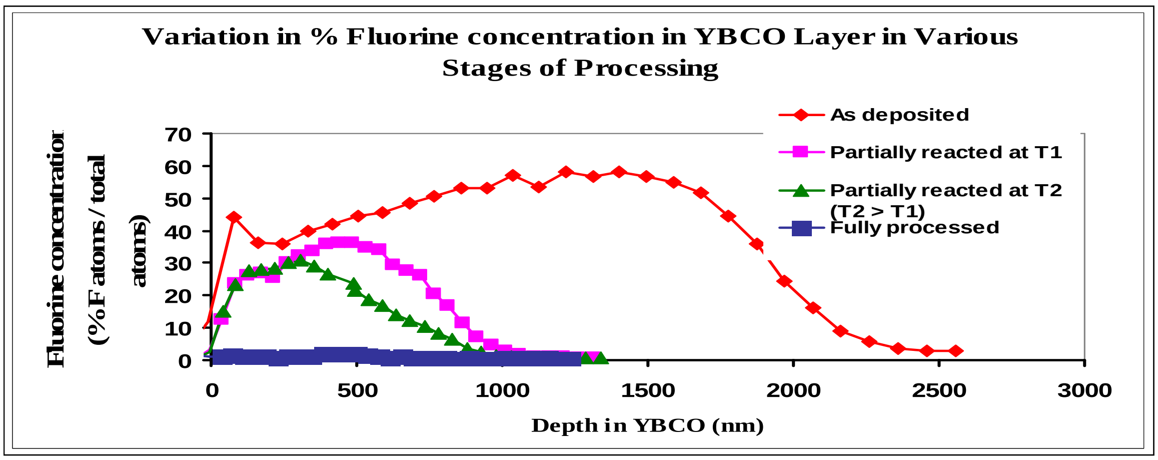

Figure 3: Variation in % Fluorine concentration in YBCO layer in various stages of processing

Table 1: Summary of fluorine concentration from Nuclear Reaction Analysis

Sample information	YBCO thickness FIB (nm) ± 30 nm	Max F position (nm) ± 50 nm	Depth of F in YBCO (nm) ± 50 nm	Max % Fluorine concentration
As deposited	2290	1300	1900	58
Partially reacted at T_1	1530	500	800	36
Partially reacted at T_2 ($T_2 > T_1$)	1460	300	600	30
Fully processed at T_2	1460	-	-	< 1.5%

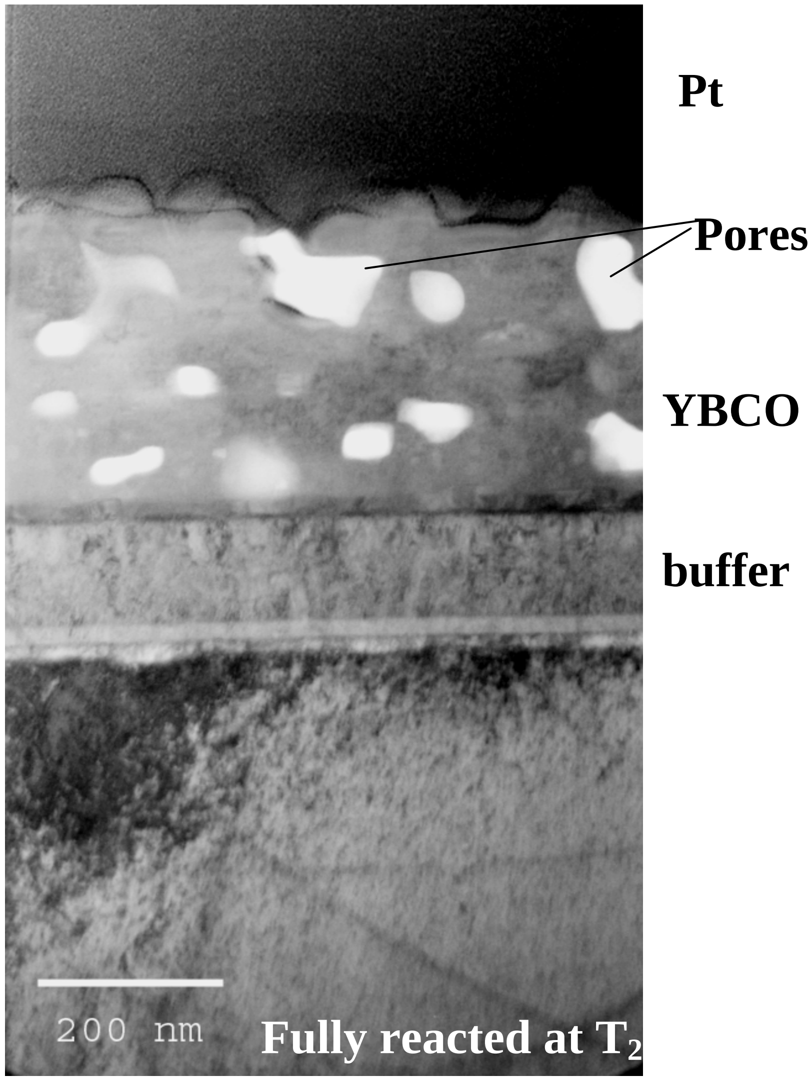

Figure 4: Bright field TEM image of fully reacted sample

As per NRA technique, the fluorine removal proceeds at the YBCO/buffer interface and the percentage fluorine was found to decrease from 58% for the partially reacted sample to less than 1.5 % for the fully processed film. TEM analysis confirms existence of pores in fully reacted sample. We believe there is scope for further process optimization for densification. The fully optimized YBCO sample, ~1.46 mm thick, carried I_c = 200 A, J_c ~ 2.5 MA/cm^2, and had less than 1.5% fluorine.

In collaboration with Oak Ridge National Laboratory, Albany NanoTech developed novel rare earth buffer, synthesized using all solution technique for high density YBCO coated conductors. The low cost chemical solution deposition (CSD) technique has been employed to synthesize textured Europium Niobate, Eu_3NbO_7 (ENO) based buffer layers, for growing thick superconducting YBCO films, on biaxially textured metal substrate. The buffer layers were characterized using RBS for compositional analysis, Secondary Ion Mass Spectroscopy (SIMS) and XPS for elemental depth profiling, FIB and SEM for cross sectional and microstructural evaluation.

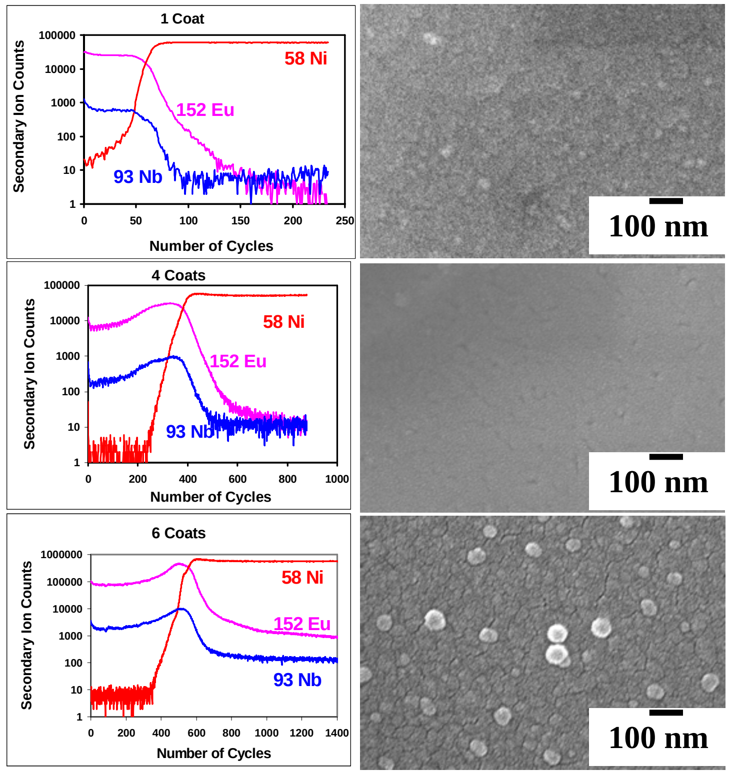

Figure 5: SIMS depth profile and corresponding microstructure of samples with 1 coat, 4 coats and 6 coats of Eu3NbO7. The depth profile shows uniform and continuous Eu and Nb distribution, and no migration of Ni into the buffer layer for 4 and 6 coat samples. The microstructure of 1 coat, 4 coats and 6 coats samples appear very different.

Epitaxial Eu_3NbO_7 buffers have been grown using CSD technique on biaxially textured Ni-W substrates. Smooth and well distinct buffer cross section observed in both 4 and 6-coat samples. Typical total ENO film thickness ~ 217 ± 30 nm (from FIB). SIMS analysis on these samples showed the presence of broad interface and diffusion of Ni into the film. The FIB images showed diffused interface between the buffer and substrate for all the three samples, confirming the observation of Ni diffusion from SIMS. The SEM images showed uniform distribution of sub micron size particles through out the samples. Ni diffusion is contained within 100 nm of ENO. YBCO films with a J_c of 800,000 A/cm^2 at 77 K and self-field have been grown on ENO buffered Ni-W substrates using pulsed laser deposition. ENO can potentially be used as a starting template for growing all solution buffers and superconductors.